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(54) METHOD FOR SOLIDIFICATION TREATMENT OF USED MERCURY ADSORBENT

(57)Abstract:

PROBLEM TO BE SOLVED: To solidify a used mercury adsorbent in a stabilized state by solidifying a used mercury adsorbent having adsorbed mercury, using a solidifying material composed of a mixture of Portland cement with blastfurance slag.

SOLUTION: In the case of solidification treatment of a used mercury adsorbent A having been used in an adsorption removal treatment process of mercury in a natural gas condensate, at first a water content rate of the used mercury adsorbent A is adjusted. In the case where the mercury adsorbent A is dry in this case, a specific amount of water is added, and further when the mercury adsorbent A is dipped in water, draining is executed. Then, a solidifying material D prepared by mixing a granulated blast furnace slag C, a Portland cement B or a previously prepared blast furnace cement D as the solidifying material is added to the marcury adsorbent A, and kneaded. After kneading, the kneaded material is charged into a solidification container and allowed to make a solidified material by curing for about

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one month. Further, a formulation ratio of the blastfurnace slag C and the Portland cement B is preferably in the range of 10/90 to 80/20 in wt. ratio.

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CLAIMS

[Claim(s)]

[Claim 1]A solidification method of used mercury adsorbent carrying out solidification of the used mercury adsorbent which adsorbed mercury using a solidifying material which mixes portland cement and blast furnace slag.

[Claim 2]A solidification method of the used mercury adsorbent according to claim 1 making into the range of 10 / 90 - 80/20 a blending ratio of blast furnace slag and portland cement which constitute a solidifying material by a weight ratio.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]

[Field of the Invention]In this invention, it is related with the solidification method of used mercury adsorbent.

In detail Therefore, gaseous hydrocarbon, such as natural gas, or LNG (liquefied natural gas), It is related with the solidification method of the used mercury adsorbent used for the adsorption treatment treatment process of mercury in liquid hydrocarbon, such as NGL (natural gas liquid), kerosene, gas oil, gasoline, and naphtha, especially a natural GASUKONDENSE rate.

[0002]

[Description of the Prior Art]The mercury contained in natural gas, a natural gas liquid (NGL), etc. has a problem of the corrosion of a heat exchanger made of aluminum, and the performance degradation of a catalyst.

[0003] Then, the art which carries out adsorption treatment of the mercury contained in natural gas, a natural gas liquid, etc. using mercury adsorbent is provided.

[0004]In mercury adsorbent, it is alumina support. There are nickel (nickel), Co (cobalt), a thing that added Mo (molybdenum) sulfide, a thing which comprises an activated carbon independent, a thing which supported a sulfuration alkaline metal and sulfuration alkaline—earth metals to the activated carbon simple substance further, etc.

[0005]On the other hand, since mercury adsorbent after adsorbing mercury is the obnoxious waste having contained mercury, it must be processed by a safe method. After also driving out used mercury adsorbent as mercury vapour with heating, the method of making condense and processing as metal mercury is safe for it. [as well as a dry cell, a fluorescent lamp, etc.] However, even if Japan does not have small deer disposal equipment and it sees globally, there are few the numbers and there are especially in Southeast Asia. [no] This is because the operation management of equipment is [that disposal equipment is expensive and] difficult. The method of solidifying into cement etc. and disposing of appropriately as what is replaced with this method, is mentioned. [0006]

[Problem(s) to be Solved by the Invention] by the way, common poisonous metal — for example, — Some methods of carrying out cement solidification processing of Cd (cadmium), Pb (lead), Hg (mercury), the Cr (chromium), etc. are proposed.

[0007] For example, Provisional Publication No. In No. 133578 [53 to] ("solidifying method by cement of chromic compound content waste"). By carrying out addition mixing of hydraulic cement, blast furnace slag powder, ferrous sulfate, or the ferrous chloride, even after hydration of cement advances, the art of acquiring the cement solidification object which is not put to acid conditions and was excellent in airtightness and watertightness is indicated.

[0008] Publication number In No. 10739 [eight to] ("waste treatment material and waste disposal

method"). The burned ash waste containing poisonous metal, such as mercury, lead, and cadmium, an acid earth, The art which carries out solidification by bentonite, kaolin, aluminum sulfate, aluminium silicate, the solid acid of a silicon dioxide or this solid acid, cement, and the thing that added the anti-caking agent to this further is indicated considering the elution prevention art of Pb as a center.

[0009] However, since it was an inorganic compound and an organic compound, in order to have processed used mercury adsorbent stably, the conventional cement solidification processing of the chemical form voice of the mercury by which used mercury adsorbent was adsorbed was insufficient.

[0010] That is, in used mercury adsorbent, mercury is adsorbed mainly with the gestalt of mercury sulfide, and while this mercury sulfide is insolubility and has stable character to water, in an oxidizing atmosphere, it shows the character which oxidizes to sulfate and is eluted easily.

[0011] Since a kind of mercury adsorbent has the character which reacts to alkali and is dissolved under a strong-base condition, When the ordinary portland cement in which strong alkali is shown according to a solidification process, and the alkali stimulus solidifying material which uses sodium hydroxide as a stimulant are used, and mercury adsorbent dissolves in a solidification process, there is inconvenience that solidification is checked and intensity is not revealed.

[0012]It is in the purpose of this invention providing the solidification method of used mercury adsorbent which can carry out solidification of the used mercury adsorbent which adsorbed mercury in the state where it was stabilized, in view of the above-mentioned actual condition.
[0013]

[Means for Solving the Problem] There are few alkali yields produced in a solidification process, and a solidifying material which has the capability to stabilize mercury sulfide because the solidifying material itself shows reduction nature is ideal for controlling a problem in cement solidification processing of the **** former mentioned above.

[0014]An artificer of this application found out that what is called blast furnace cement that mixes portland cement and blast furnace slag was the optimal as a solidifying material with which it is satisfied of conditions like ****.

[0015]So, in a solidification method of used mercury adsorbent in connection with this invention, solidification of the used mercury adsorbent which adsorbed mercury is carried out using a solidifying material which mixes portland cement and blast furnace slag in order to attain the above-mentioned purpose.

[0016]

[Embodiment of the Invention] <u>Drawing 1</u> shows the example which applied the solidification method of the used mercury adsorbent in connection with this invention to the adsorption treatment treatment process which removes mercury out of a natural GASUKONDENSE rate (NGL).

[0017]As shown in <u>drawing 1</u>, the solidification of used mercury adsorbent includes the process (S2) of kneading the process (S1) of adjusting the moisture regain of used mercury adsorbent, and used mercury adsorbent and a solidifying material, and the process (S2) of feeding the kneaded material of used mercury adsorbent and a solidifying material into a solidification container.

[0018]In order to carry out solidification of the used mercury adsorbent like ****, the moisture regain of the used mercury adsorbent A is adjusted first (S1). The "mercury adsorbent" under following explanation shall point out "used mercury adsorbent."

[0019] Here, when the mercury adsorbent A is dry, the water of the specified quantity is added to this mercury adsorbent A, and when the mercury adsorbent A is immersed in water, the moisture regain of the mercury adsorbent A is adjusted by draining off water to the mercury adsorbent A. At the time of adjustment of this moisture regain, the mercury adsorbent A may still be the fabricated gestalt, or may be ground.

[0020] After adjusting the moisture regain of the mercury adsorbent A, in addition to the mercury adsorbent A with which moisture regain was adjusted, blast furnace cement D as the solidifying

material D which mixes blast-furnace-slag B and the portland cement C, or a solidifying material prepared beforehand is kneaded (S2).

[0021]When mixing the solidifying material D with the mercury adsorbent A, the mercury adsorbent A with which moisture regain was adjusted to the cement base (solidifying material) D which added, kneaded and prepared water to the solidifying material D which mixes blast-furnace-slag B and the portland cement C, or blast furnace cement may be added.

[0022] After kneading the mercury adsorbent A and the solidifying material (blast furnace cement) D, this kneaded material is fed into the solidification container (not shown) of specified shape (S3). After this, by recuperating oneself about one month, solidification of the mercury adsorbent A will be carried out because the mercury adsorbent A and kneaded material with the solidifying material D change with a solidified body.

[0023] As stated also in advance, mercury adsorbent adsorbs mercury as mercury sulfide here, As a method of solidifying so that elution of mercury from hydrogen adsorbent may be controlled and disposal may be suited, application of the cement solidification material which blended blast furnace slag is the optimal, without having the capability to insolubilize and spoiling this capability. [0024] By namely, the thing for which mercury adsorbent is solidified with the cement which iron and the sulfur component which show reduction nature contain in blast furnace slag, and blended blast furnace slag. Since mercury adsorbent can be solidified in reducing atmosphere, it becomes possible to fix stably with the gestalt of mercury sulfide which is an adsorption gestalt of mercury. [0025] The mercury adsorbent containing nickel, Co, and Mo, When the solidifying material which generates a lot of [since nickel etc. dissolve under a strong-base condition / when solidifying like portland cement] calcium hydroxide is used, Solidification of cement is checked with the metal which the mercury fixed performance which some mercury adsorbent dissolves and mercury adsorbent has was spoiled, and dissolved. In order to suppress the dissolution of such mercury adsorbent and to generate a stable solidified body, an alkaline low solidifying material is suitable and the cement solidification material which contains blast furnace slag so much is suitable as an alkaline low solidifying material.

[0026] Therefore, according to the fixing method of the used mercury adsorbent in connection with this invention which carries out solidification of the mercury adsorbent using the solidifying material which mixes portland cement and blast furnace slag as mentioned above. It becomes possible to fix used mercury adsorbent in the state where it was stabilized, without causing the insufficient strength of unprepared elution of mercury, or a solidified body, since the thing which fix stably the mercury by which mercury adsorbent was adsorbed and for which solidification of a solidifying material is prevented from alkaline lowness while being able to carry out things can be suppressed. [0027] Since the solidifying material which mixes portland cement and blast furnace slag is generally cheaply marketed as blast furnace cement and can be easily obtained not only in the inside of Japan but in every country in the world, it becomes possible to carry out solidification of the used mercury adsorbent simple and cheaply.

[0028]In the fixing method of the used mercury adsorbent in connection with this invention, the main work, In order to mix [very easy] used mercury adsorbent, a solidifying material, and water, it becomes possible to carry out solidification of the used mercury adsorbent very easily into the petroleum plant which used mercury adsorbent generates.

[0029] Table 1 indicated to drawing 2 expresses the result of having carried out the elution test, to the solidified body created using various kinds of solidifying materials.

[0030]the so-called pellet of the nickel-Cr-MoX catalyst which made the gamma alumina carrier support nickel, Cr, and Mo sulfide with this examination — mercury 0.8 wt% — the thing made to adsorb is made into the sample of mercury adsorbent.

[0031]The comparative examination is carried out about three sorts of solidified bodies and the mercury adsorbent simple substance which were created using these solidifying materials, using an ordinary-portland-cement and blast furnace cement C kind equivalent (portland cement/blast

furnace slag = 35/65 wt%) and slag cement as a solidifying material.

[0032]Each solidified body is mercury adsorbent. It is water to 5 g. 9.2 g, solidifying material After adding 17.9 g and mixing, this was supplied to the solidification container, and in the room temperature, care of health was performed for one month, and it created. In using slag cement as a solidifying material, it has also added the sodium hydroxide solution as a hardening agent. [0033]The elution test was carried out according to the "assaying method of metal etc. which are contained in industrial waste" Environment-Protection-Agency notification No. (February, Showa 48) 13. That is, a sample is extracted from liquid after grinding the created solidified body and shaking in weak chloride aqueous acids for ordinary temperature 6 hours, and this sample is quantified by hydride generation atomic absorption spectrophotometry.

[0034] The solidified body using blast furnace cement as a solidifying material has the low mercury concentration in an eluate compared with a mercury adsorbent simple substance, and it is clear from the test result of Table 1 that its elution of mercury is controlled by solidification.

[0035]On the other hand, in the solidified body using portland cement as a solidifying material, and the solidified body using slag cement as a solidifying material, the phenomenon in which elution of mercury will be promoted conversely is seen compared with a mercury adsorbent simple substance. [0036]On the other hand, it turns out that the oxidation-reduction potential of the eluate shows the low value in the solidified body which used blast furnace cement and slag cement as the solidifying material, and these solidified bodies show reduction nature.

[0037] However, although the solidified body which used slag cement as the solidifying material has a low oxidation-reduction potential, since the sodium hydroxide solution is added as a hardening agent, the dissolution of mercury adsorbent arises, and it does not solidify thoroughly, but, therefore, the elution depressor effect of mercury will become small.

[0038] When used mercury adsorbent is fixed from this result, controlling elution of mercury effectively, it turns out that use of the solidifying material which blends portland cement and blast furnace slag is the optimal.

[0039] Table 2 indicated to drawing 3 expresses the result of having carried out the elution test, to the solidified body created using the various solidifying materials which changed the mixture ratio of portland cement and blast furnace slag.

[0040]In this examination, the chemical form voice of the mercury by which mercury adsorbent was adsorbed is imitated, and the powder of mercury sulfide is used as a sample.

[0041]What mixed three sorts of blast furnace slag from which a maker differs, and portland cement by various kinds of weight ratios of 5/95, 10/90, 20/80, 40/60, 65/35, and 80/20 is used as a solidifying material.

[0042]In a solidified body and water of 14.3 g A 35.7-g solidifying material is added, and it mixes, and is this. After adding 0.058-g mercury sulfide powder and mixing, it is creating by supplying to a solidification container and recuperating oneself.

[0043]According to the "assaying method of metal etc. which are contained in industrial waste" Environment-Protection-Agency notification No. (February, Showa 48) 13, an elution test as well as the examination which showed the result in Table 1, A sample is extracted from liquid after grinding the created solidified body and shaking in weak chloride aqueous acids for ordinary temperature 6 hours, and this sample is quantified by hydride generation atomic absorption spectrophotometry. [0044]The mercury concentration in the eluate in each solidified body is not based on the difference in the maker of blast furnace slag, but shows the almost same measured value so that clearly from Table 2. It turns out that it is what is revealed based on the physical properties with which the fixed effect of mercury in the solidifying material having contained blast furnace slag is not based on a specific kind of blast furnace slag, and blast furnace slag is generally provided from this. [0045]When the blending ratio of blast furnace slag to portland cement uses a low solidifying material, Although the tendency for the elution volume of mercury to increase is seen, the blending ratio of blast furnace slag and portland cement is a weight ratio. In 10/90 – 80/20, the elution

volume of mercury shows the low value.

[0046]As a result, when used mercury adsorbent is fixed controlling elution of mercury effectively, the blending ratio of blast furnace slag and portland cement is a weight ratio. It turns out that the solidifying material in the range of 10 / 90 - 80/20 can be used.

[0047]The solidification method of the mercury adsorbent in connection with this invention, Only not only in the adsorption treatment treatment process which removes mercury out of a natural GASUKONDENSE rate (NGL), Also when removing mercury out of liquid hydrocarbon, such as gaseous hydrocarbon, such as natural gas, LNG (liquefied natural gas), kerosene, gas oil, gasoline, naphtha, it cannot be overemphasized that it can apply very effectively.

[0048]

[Effect of the Invention] As mentioned above, as explained in full detail, in the solidification method of the used mercury adsorbent in connection with this invention, solidification of the used mercury adsorbent which adsorbed mercury is carried out using the solidifying material which mixes portland cement and blast furnace slag.

[0049]according to the fixing method of the used mercury adsorbent in connection with this invention, the mercury by which mercury adsorbent was adsorbed is fixed stably, while being able to carry out things, It becomes possible to fix used mercury adsorbent in the state where it was stabilized, without causing the insufficient strength of unprepared elution of mercury, or a solidified body, since it can suppress that solidification of a solidifying material is prevented from alkaline lowness.

[0050] The solidifying material which mixes portland cement and blast furnace slag according to the fixing method of the used mercury adsorbent in connection with this invention, Since it is generally cheaply marketed as blast furnace cement and not only the inside of Japan but every country in the world can be obtained easily, it becomes possible to carry out solidification of the used mercury adsorbent simple and cheaply.

[0051]In order for main work to mix [very easy] used mercury adsorbent, a solidifying material, and water according to the fixing method of the used mercury adsorbent in connection with this invention, Even if it is in the petroleum plant which used mercury adsorbent generates, it becomes possible to carry out solidification of the used mercury adsorbent very easily.

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TECHNICAL FIELD

[Field of the Invention]In this invention, it is related with the solidification method of used mercury adsorbent.

In detail Therefore, gaseous hydrocarbon, such as natural gas, or LNG (liquefied natural gas), It is related with the solidification method of the used mercury adsorbent used for the adsorption treatment treatment process of mercury in liquid hydrocarbon, such as NGL (natural gas liquid), kerosene, gas oil, gasoline, and naphtha, especially a natural GASUKONDENSE rate.

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EFFECT OF THE INVENTION

[Effect of the Invention] As mentioned above, as explained in full detail, in the solidification method of the used mercury adsorbent in connection with this invention, solidification of the used mercury adsorbent which adsorbed mercury is carried out using the solidifying material which mixes portland cement and blast furnace slag.

[0049] according to the fixing method of the used mercury adsorbent in connection with this invention, the mercury by which mercury adsorbent was adsorbed is fixed stably, while being able to carry out things, It becomes possible to fix used mercury adsorbent in the state where it was stabilized, without causing the insufficient strength of unprepared elution of mercury, or a solidified body, since it can suppress that solidification of a solidifying material is prevented from alkaline lowness.

[0050]According to the fixing method of the used mercury adsorbent in connection with this invention, generally, the solidifying material which mixes portland cement and blast furnace slag is cheaply marketed as blast furnace cement.

Since not only the inside of Japan but every country in the world can be obtained easily, it becomes possible to carry out solidification of the used mercury adsorbent simple and cheaply.

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[0009]However, since it was an inorganic compound and an organic compound, in order to have processed used mercury adsorbent stably, the conventional cement solidification processing of the chemical form voice of the mercury by which used mercury adsorbent was adsorbed was insufficient.

[0010] That is, in used mercury adsorbent, mercury is adsorbed mainly with the gestalt of mercury sulfide, and while this mercury sulfide is insolubility and has stable character to water, in an oxidizing atmosphere, it shows the character which oxidizes to sulfate and is eluted easily.

[0011] Since a kind of mercury adsorbent has the character which reacts to alkali and is dissolved under a strong-base condition, When the ordinary portland cement in which strong alkali is shown according to a solidification process, and the alkali stimulus solidifying material which uses sodium hydroxide as a stimulant are used, and mercury adsorbent dissolves in a solidification process, there is inconvenience that solidification is checked and intensity is not revealed.

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MEANS

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[0014]An artificer of this application found out that what is called blast furnace cement that mixes portland cement and blast furnace slag was the optimal as a solidifying material with which it is satisfied of conditions like ****.

[0015]So, in a solidification method of used mercury adsorbent in connection with this invention, solidification of the used mercury adsorbent which adsorbed mercury is carried out using a solidifying material which mixes portland cement and blast furnace slag in order to attain the abovementioned purpose.

[0016]

[Embodiment of the Invention] <u>Drawing 1</u> shows the example which applied the solidification method of the used mercury adsorbent in connection with this invention to the adsorption treatment treatment process which removes mercury out of a natural GASUKONDENSE rate (NGL). [0017] As shown in <u>drawing 1</u>, the solidification of used mercury adsorbent includes the process (S2) of kneading the process (S1) of adjusting the moisture regain of used mercury adsorbent, and used mercury adsorbent and a solidifying material, and the process (S2) of feeding the kneaded material of used mercury adsorbent and a solidifying material into a solidification container. [0018] In order to carry out solidification of the used mercury adsorbent like ****, the moisture

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[0019] Here, when the mercury adsorbent A is dry, the water of the specified quantity is added to this mercury adsorbent A, and when the mercury adsorbent A is immersed in water, the moisture regain of the mercury adsorbent A is adjusted by draining off water to the mercury adsorbent A. At the time of adjustment of this moisture regain, the mercury adsorbent A may still be the fabricated gestalt, or may be ground.

[0020] After adjusting the moisture regain of the mercury adsorbent A, in addition to the mercury adsorbent A with which moisture regain was adjusted, blast furnace cement D as the solidifying material D which mixes blast-furnace-slag B and the portland cement C, or a solidifying material prepared beforehand is kneaded (S2).

[0021]When mixing the solidifying material D with the mercury adsorbent A, the mercury adsorbent A with which moisture regain was adjusted to the cement base (solidifying material) D which added, kneaded and prepared water to the solidifying material D which mixes blast-furnace-slag B and the portland cement C, or blast furnace cement may be added.

[0022] After kneading the mercury adsorbent A and the solidifying material (blast furnace cement) D, this kneaded material is fed into the solidification container (not shown) of specified shape (S3).

After this, by recuperating oneself about one month, solidification of the mercury adsorbent A will be carried out because the mercury adsorbent A and kneaded material with the solidifying material D change with a solidified body.

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[0026]Therefore, according to the fixing method of the used mercury adsorbent in connection with this invention which carries out solidification of the mercury adsorbent using the solidifying material which mixes portland cement and blast furnace slag as mentioned above. It becomes possible to fix used mercury adsorbent in the state where it was stabilized, without causing the insufficient strength of unprepared elution of mercury, or a solidified body, since the thing which fix stably the mercury by which mercury adsorbent was adsorbed and for which solidification of a solidifying material is prevented from alkaline lowness while being able to carry out things can be suppressed. [0027]Since the solidifying material which mixes portland cement and blast furnace slag is generally cheaply marketed as blast furnace cement and can be easily obtained not only in the inside of Japan but in every country in the world, it becomes possible to carry out solidification of the used mercury adsorbent simple and cheaply.

[0028]In the fixing method of the used mercury adsorbent in connection with this invention, the main work, In order to mix [very easy] used mercury adsorbent, a solidifying material, and water, it becomes possible to carry out solidification of the used mercury adsorbent very easily into the petroleum plant which used mercury adsorbent generates.

[0029] Table 1 indicated to drawing 2 expresses the result of having carried out the elution test, to the solidified body created using various kinds of solidifying materials.

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[0031]The comparative examination is carried out about three sorts of solidified bodies and the mercury adsorbent simple substance which were created using these solidifying materials, using an ordinary-portland-cement and blast furnace cement C kind equivalent (portland cement/blast furnace slag = 35/65 wt%) and slag cement as a solidifying material.

[0032]Each solidified body is mercury adsorbent. It is water to 5 g. 9.2 g, solidifying material After adding 17.9 g and mixing, this was supplied to the solidification container, and in the room temperature, care of health was performed for one month, and it created. In using slag cement as a solidifying material, it has also added the sodium hydroxide solution as a hardening agent.
[0033]The elution test was carried out according to the "assaying method of metal etc. which are contained in industrial waste" Environment-Protection-Agency notification No. (February, Showa 48) 13. That is, a sample is extracted from liquid after grinding the created solidified body and

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[0034] The solidified body using blast furnace cement as a solidifying material has the low mercury concentration in an eluate compared with a mercury adsorbent simple substance, and it is clear from the test result of Table 1 that its elution of mercury is controlled by solidification.

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[0037] However, although the solidified body which used slag cement as the solidifying material has a low oxidation-reduction potential, since the sodium hydroxide solution is added as a hardening agent, the dissolution of mercury adsorbent arises, and it does not solidify thoroughly, but, therefore, the elution depressor effect of mercury will become small.

[0038]When used mercury adsorbent is fixed from this result, controlling elution of mercury effectively, it turns out that use of the solidifying material which blends portland cement and blast furnace slag is the optimal.

[0039] Table 2 indicated to <u>drawing 3</u> expresses the result of having carried out the elution test, to the solidified body created using the various solidifying materials which changed the mixture ratio of portland cement and blast furnace slag.

[0040]In this examination, the chemical form voice of the mercury by which mercury adsorbent was adsorbed is imitated, and the powder of mercury sulfide is used as a sample.

[0041]What mixed three sorts of blast furnace slag from which a maker differs, and portland cement by various kinds of weight ratios of 5/95, 10/90, 20/80, 40/60, 65/35, and 80/20 is used as a solidifying material.

[0042]In a solidified body and water of 14.3 g A 35.7-g solidifying material is added, and it mixes, and is this. After adding 0.058-g mercury sulfide powder and mixing, it is creating by supplying to a solidification container and recuperating oneself.

[0043] According to the "assaying method of metal etc. which are contained in industrial waste" Environment–Protection–Agency notification No. (February, Showa 48) 13, an elution test as well as the examination which showed the result in Table 1, A sample is extracted from liquid after grinding the created solidified body and shaking in weak chloride aqueous acids for ordinary temperature 6 hours, and this sample is quantified by hydride generation atomic absorption spectrophotometry. [0044] The mercury concentration in the eluate in each solidified body is not based on the difference in the maker of blast furnace slag, but shows the almost same measured value so that clearly from Table 2. It turns out that it is what is revealed based on the physical properties with which the fixed effect of mercury in the solidifying material having contained blast furnace slag is not based on a specific kind of blast furnace slag, and blast furnace slag is generally provided from this. [0045] When the blending ratio of blast furnace slag to portland cement uses a low solidifying material, Although the tendency for the elution volume of mercury to increase is seen, the blending ratio of blast furnace slag and portland cement is a weight ratio. In 10/90 – 80/20, the elution volume of mercury shows the low value.

[0046]As a result, when used mercury adsorbent is fixed controlling elution of mercury effectively, the blending ratio of blast furnace slag and portland cement is a weight ratio. It turns out that the solidifying material in the range of 10 / 90 - 80/20 can be used.

[0047] The solidification method of the mercury adsorbent in connection with this invention, Only not only in the adsorption treatment treatment process which removes mercury out of a natural GASUKONDENSE rate (NGL), Also when removing mercury out of liquid hydrocarbon, such as gaseous hydrocarbon, such as natural gas, LNG (liquefied natural gas), kerosene, gas oil, gasoline,

naphtha, it cannot be overemphasized that it can apply very effectively.

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] The work-flows figure showing the solidification method of the used mercury adsorbent in connection with this invention.

[Drawing 2] The table 1 showing the result of having carried out the elution test to the solidified body created using various kinds of solidifying materials.

[Drawing 3] The table 2 showing the result of having carried out the elution test to the solidified body created using the various solidifying materials which changed the mixing ratio of portland cement and blast furnace slag.

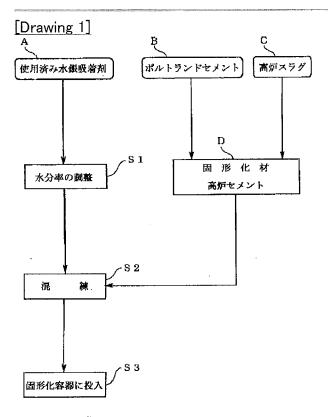
[Description of Notations]

- A -- Used mercury adsorbent,
- B -- Portland cement,
- C -- Blast furnace slag
- D -- A solidifying material, blast furnace cement.

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DRAWINGS



[Drawing 2]

表	

固化体(固化材)	溶出液中の水銀濃度	溶出液の酸化還元電位
水銀吸着剤単体	1. 5 ppb	Eh = 140 mV
ポルトラントセメント	16.5 ррт	Eh = 50 mV
高炉セメント	< 0, 5 ppb	Eh = −93 mV
スラグセメント	145 ppb	Eh = -106 mV

[Drawing 3]

表 2

固化体(固化材)	高炉スラグ	溶出液中の
(高炉スラグ/ボルトランドセメント 比)	メーカー	水銀濃度(ppm)
5/95 (vrt%)	A社	15
10/90 (wt%)	A社	1
	A社	0. 5
20/80 (wt%)	B社	< 0. 1
	C社	< 0. 1
	A社	0.3
40/60 (wt%)	B社	< 0. 1
	C社	< 0. 1
65/35 (wt%)	A社	< 0. 1
	B牡	< 0. 1
	C社.	< 0. 1
80/20 (wt%)	A社	< 0. 1

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- PN JP11347516 A 19991221
- TI METHOD FOR SOLIDIFICATION TREATMENT OF USED MERCURY ADSORBENT
- FI B09B3/00+ZAB : B09B3/00&301K : B09B3/00&301S : C04B7/19
- PA JGC CORP
- IN SASAKI TADASHI; SATO KAZUO; FUJIMURA YASUSHI; YAMADA MASATOSHI
- AP JP19980162180 19980610
- PR JP19980162180 19980610
- DT 1

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- AN 2000-110248 [10]
- Solidification processing of used absorbent for mercury in liquid and gaseous hydrocarbon compounds involves using mixture of portland cement and blast furnace slag as solidification material
- AB JP11347516 NOVELTY The solidification material for absorbing portland, contains a mixture of used mercury cement and blast furnace slag.
 - USE For solidification of used absorbent of mercury in liquid and gaseous hydrocarbons such as liquid natural gas, natural gas liquid, kerosene, light oil, gasoline, naphtha etc.
 - ADVANTAGE Solidification of used absorbent of mercury is carried out cheaply.
 - (Dwg.0/3)
- W SOLIDIFICATION PROCESS ABSORB MERCURY LIQUID GAS HYDROCARBON COMPOUND MIXTURE PORTLAND CEMENT BLAST FURNACE SLAG SOLIDIFICATION MATERIAL
- PN JP11347516 A 19991221 DW200010 B09B3/00 005pp
- IC B09B3/00; C04B7/19
- PA (JAGA) JGC CORP

@ PAJ / JPO

- PN JP11347516 A 19991221
- TI METHOD FOR SOLIDIFICATION TREATMENT OF USED MERCURY ADSORBENT
 - PROBLEM TO BE SOLVED: To solidify a used mercury adsorbent in a stabilized state by solidifying a
 used mercury adsorbent having adsorbed mercury, using a solidifying material composed of a mixture
 of Portland cement with blastfurance slag.
 - SOLUTION: In the case of solidification treatment of a used mercury adsorbent A having been used in an adsorption removal treatment process of mercury in a natural gas condensate, at first a water content rate of the used mercury adsorbent A is adjusted. In the case where the mercury adsorbent A is dry in this case, a specific amount of water is added, and further when the mercury adsorbent A is dipped in water, draining is executed. Then, a solidifying material D prepared by mixing a granulated blast furnace slag C, a Portland cement B or a previously prepared blast furnace cement D as the solidifying material is added to the marcury adsorbent A, and kneaded. After kneading, the kneaded material is charged into a solidification container and allowed to make a solidified material by curing for about one month. Further, a formulation ratio of the blastfurnace slag C and the Portland cement B is preferably in the range of 10/90 to 80/20 in wt. ratio.
- B09B3/00 ;B09B3/00
- st C04B7/19
- PA JGC CORP
- IN FUJIMURA YASUSHI;SASAKI TADASHI;YAMADA MASATOSHI;SATO KAZUO
- ABD 20000330
- ABV 200003
- AP JP19980162180 19980610
- PD 1999-12-21

(19)日本国特許庁(JP)

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(11)特許出願公開番号

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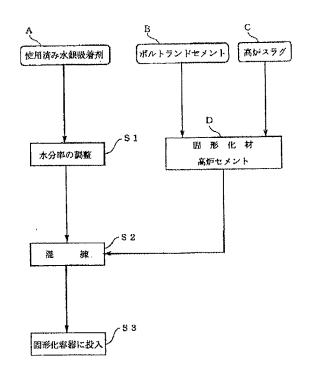
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(22)出願日	平成10年(1998) 6月10日	東京都千代田区大手町2丁目2番1号
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		最終質に続く

(54) 【発明の名称】 使用済み水銀吸着剤の固化処理方法

(57)【要約】

【課題】 本発明の課題は、水銀を吸着した使用済みの水銀吸着剤を、水銀の溶出を抑制して安定に固化処理することの可能な、使用済み水銀吸着剤の固化処理方法を提供することにある。

【解決手段】 本発明に関わる使用済み水銀吸着剤の固化処理方法は、ボルトランドセメントと高炉スラグとを混合して成る固化材を用いて、水銀を吸着した使用済み水銀吸着剤を固化処理している。



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【特許請求の範囲】

ポルトランドセメントと高炉スラグと 【請求項1】 を混合して成る固化材を用いて、水銀を吸着した使用済 み水銀吸着剤を固化処理することを特徴とする使用済み 水銀吸着剤の固化処理方法。

固化材を構成する高炉スラグとポルト 【請求項2】 ランドセメントとの配合割合を、重量比で10/90~ 80/20の範囲としたことを特徴とする請求項1記載 の使用済み水銀吸着剤の固化処理方法。

【発明の詳細な説明】

[0001]

【発明の属する技術分野】本発明は、使用済み水銀吸着 剤の固化処理方法に関するもので、詳しくは、天然ガス 等のガス状炭化水素、あるいはLNG(液化天然ガス)、 NGL(天然ガスリキッド)、灯油、軽油、ガソリン、ナ フサ等の液状炭化水素、特に天然ガスコンデンセレート 中の水銀の吸着除去処理プロセスに用いられた使用済み の水銀吸着剤の固化処理方法に関する。

[0002]

【従来の技術】天然ガスや天然ガスリキッド(NGL)等 20 に含まれている水銀は、アルミニウム製熱交換器の腐食 や、触媒の性能劣化の問題がある。

【0003】そこで、天然ガスや天然ガスリキッド等に 含まれている水銀を、水銀吸着剤を用いて吸着除去する 技術が提供されている。

【0004】水銀吸着剤には、アルミナ担持体に Ni (ニッケル)、Co(コバルト)、Mo(モリブデン)硫化物 を添加したもの、活性炭単独から成るもの、さらには活 性炭単体に硫化アルカリ金属や硫化アルカリ土類金属を 担持したもの等がある。

【〇〇〇5】一方、水銀を吸着した後の水銀吸着剤は、 水銀を含んだ有害な廃棄物であるため、安全な方法で処 理しなければならない。使用済み水銀吸着剤も、乾電池 や蛍光灯等と同様、加熱により水銀蒸気として追い出し たあと、凝縮させて金属水銀として処理する方法が安全 である。しかし、日本には僅かしか処理設備がなく、ま た世界的に見てもその数は少なく、特に東南アジア等で は皆無である。これは処理設備が高価なこと、および設 備の運転管理が難しいことによる。この方法に代わるも のとして、セメント等で固形化して適切に処分する方法 が挙げられる。

[0006]

【発明が解決しようとする課題】ところで、一般的な有 害金属、例えば Cd(カドミウム)、Pb(鉛)、Hg(水 銀)、Cr(クロム)等を、セメント固化処理する方法が幾 つか提案されている。

【0007】例えば、特開昭 53-133578号(「三価クロ ム化合物含有廃棄物のセメントによる固化方法」)に は、水硬性セメントと高炉水滓スラグ粉末と硫酸第一鉄 ントの水和が進行した後も酸性条件に曝されることがな く気密性、水密性に優れたセメント固化体を得る技術が 開示されている。

【0008】また、特開平 8-10739号(「廃棄物処理材 及び廃棄物処理方法」)には、水銀、鉛、カドミウム等 の有害金属を含有する焼却灰廃棄物を、酸性白土、ベン トナイト、カオリン、硫酸アルミニウム、アルミニウム シリケート、または二酸化珪素の固体酸、または該固体 酸とセメント、さらにこれに固結防止剤を加えたものに 10 より固化処理する技術が、Pbの溶出防止技術を中心と して開示されている。

【〇〇〇9】しかしながら、使用済み水銀吸着剤に吸着 された水銀の化学形態は、無機化合物や有機化合物であ るため、使用済み水銀吸着剤を安定に処理するには、従 来のセメント固化処理では不十分であった。

【0010】すなわち、使用済みの水銀吸着剤におい て、水銀は主として硫化水銀の形態で吸着されており、 この硫化水銀は水に対して不溶性で、安定な性質を有し ている反面、酸化雰囲気においては、硫酸塩に酸化され て溶出し易い性質を示す。

【0011】また、水銀吸着剤の一種は、強アルカリ条 件下においてアルカリと反応して溶解する性質を有する ため、固化過程で強いアルカリを示す普通ホルトランド セメントや、水酸化ナトリウムを刺激剤として使用する アルカリ刺激固化材を使用した場合、固化過程において 水銀吸着剤が溶解することにより、固化が阻害されて強 度が発現しないという不都合が有る。

【0012】本発明の目的は上記実状に鑑みて、水銀を 吸着した使用済みの水銀吸着剤を、安定した状態で鬩化 30 処理することの可能な、使用済み水銀吸着剤の間化処理 方法を提供することにある。

[0013]

【課題を解決するための手段】上述した如き従来のセメ ント固化処理における問題を抑制するには、固化過程で 生じるアルカリ発生量が少なく、固化材自体が還元性を 示すことで硫化水銀を安定化させる能力を有する固化材 が最適である。

【0014】本願の発明者は、上述の如き条件を満足す る固化材として、ボルトランドセメントと高炉スラグと を混合して成る、いわゆる高炉セメントが最適であるこ とを見出した。

【0015】そこで、本発明に関わる使用済み水銀吸着 剤の固化処理方法では、上記目的を達成するべく、ポル トランドセメントと高炉スラグとを混合して成る固化材 を用いて、水銀を吸着した使用済み水銀吸着剤を固化処 理している。

[0016]

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【発明の実施の形態】図1は、天然ガスコンデンセレー ト(NGL)中から水銀を除去する吸着除去処理プロセス もしくは塩化第一鉄とを添加混合することにより、セメ 50 に、本発明に関わる使用済み水銀吸着剤の固化処理方法 を適用した例を示している。

【0017】図1に示すように、使用済み水銀吸着剤の 固化処理は、使用済み水銀吸着剤の水分率を調整する工 程(S1)と、使用済み水銀吸着剤と固化材とを混練する 工程(S2)と、使用済み水銀吸着剤と固化材との混練物 を固形化容器に投入する工程(S2)とを含んでいる。

【0018】上述の如く、使用済み水銀吸着剤を固化処 理するには、先ず使用済み水銀吸着剤Aの水分率を調整 する(S1)。なお、以下の説明中における「水銀吸着 剤」は、「使用済み水銀吸着剤」を指すものとする。

【0019】ここで、水銀吸着剤Aが乾燥している場合 には、この水銀吸着剤Aに所定量の水を加え、水銀吸着 剤Aが水に浸漬されている場合には、水銀吸着剤Aに対 して水切りを行うことで、水銀吸着剤Aの水分率を調整 する。なお、この水分率の調整時において、水銀吸着剤 Aは成形された形態のままであっても、あるいは粉砕し たものであっても良い。

【〇〇2〇】水銀吸着剤Aの水分率を調整したのち、高 炉スラグBとポルトランドセメントCとを混合して成る 固化材D、あるいは予め調製された固化材としての高炉 20 セメントDを、水分率の調整された水銀吸着剤Aに加え て混練する(S2)。

【0021】なお、水銀吸着剤Aと固化材Dとを混合す る際、高炉スラグBとポルトランドセメントCとを混合 して成る固化材D、あるいは高炉セメントに水を加え混 練して調製したセメントベース(固化材) Dに、水分率の 調整された水銀吸着剤Aを加えても良い。

【0022】水銀吸着剤Aと固化材(高炉セメント)Dと を混練したのち、この混練物を所定形状の固形化容器 (図示せず)に投入する(S3)。こののち、1ヶ月程度養 生することによって、水銀吸着剤Aと固化材Dとの混練 物が固化体と成ることで、水銀吸着剤Aが固化処理され ることとなる。

【0023】ここで、先にも述べたように、水銀吸着剤 は水銀を硫化水銀として吸着し、不溶化する能力を有し ており、この能力を損なうことなく、水素吸着剤からの 水銀の溶出を抑制し、処分に適合するように固形化する 方法としては、高炉スラグを配合したセメント固化材の 適用が最適である。

【0024】すなわち、高炉スラグには還元性を示す鉄 40 や硫黄成分が含有されており、高炉スラグを配合したセ メントによって水銀吸着剤を固形化することで、水銀吸 着剤を還元雰囲気中に固形化できるため、水銀の吸着形 態である硫化水銀の形態のままで安定に固定化すること が可能となる。

【0025】また、Ni、Co、Moを含む水銀吸着剤 は、強アルカリ条件下においてNi等が溶解するため、 ポルトランドセメントの如く固化する際に大量の水酸化 カルシウムを生成する固化材を使用した場合、水銀吸着 剤の一部が溶解して水銀吸着剤の持つ水銀固定化性能が 50

損なわれ、かつ溶解した金属によってセメントの固化が 阻害される。このような水銀吸着剤の溶解を抑え、安定 な固化体を生成するためには、アルカリ性の低い固化材 が適切であり、高炉スラグを多量に含むセメント固化材 は、アルカリ性の低い固化材として適切である。

【0026】したがって、上述した如くポルトランドセ メントと高炉スラグとを混合して成る固化材を用いて水 銀吸着剤を固化処理する、本発明に関わる使用済み水銀 吸着剤の固定化方法によれば、水銀吸着剤に吸着された 10 水銀を安定に固定化することできるとともに、アルカリ 性の低さから固化材の固化が阻害されることを抑えられ るので、水銀の不用意な溶出や固化体の強度不足を招く ことなく、使用済みの水銀吸着剤を安定した状態で固定 化することが可能となる。

【0027】また、ポルトランドセメントと高炉スラグ とを混合して成る固化材は、一般に高炉セメントとして 安価に市販されており、日本国内に限らず世界各国でも 容易に入手できるので、簡便かつ安価に使用済み水銀吸 着剤を固化処理することが可能となる。

【0028】さらに、本発明に関わる使用済み水銀吸着 剤の固定化方法において、その主たる作業は、使用済み 水銀吸着剤と固化材および水とを混合するという極めて 簡単なものであるため、使用済みの水銀吸着剤が発生す る石油プラント内において、使用済み水銀吸着剤を極め て容易に固化処理することが可能となる。

【0029】図2に開示した表1は、各種の固化材を用 いて作成した固化体に対し、溶出試験を実施した結果を 表している。

【0030】この試験では、ガンマアルミナ担体にN i、Cr、Mo硫化物を担持させた、いわゆるNi-Cr-Mo X 触媒のペレットに、水銀を 0.8 wt%吸着させたもの を、水銀吸着剤の試料としている。

【0031】また、固化材として普通ポルトランドセメ ント、高炉セメントC種相当品(ポルトランドセメント /高炉スラグ= 35/65 xt%)、スラグセメントを用 い、これら固化材を用いて作成した3種の固化体と水銀 吸着剤単体とについて比較検討している。

【0032】各々の固化体は、水銀吸着剤 5gに対し て、水 9.2g、固化材 17.9gを加えて混合した後、こ れを固形化容器に投入し、室温において1ヶ月養生を行 って作成した。なお、箇化材としてスラグセメントを用 いる場合には、硬化剤として水酸化ナトリウム溶液も添 加している。

【0033】また、溶出試験は「産業廃棄物に含まれる 金属等の検定方法」環境庁告示13号(昭和48年2 月) に従って実施した。すなわち、作成した固化体を粉 砕して弱塩酸酸性水溶液中で常温6時間振盪した後の液 から試料を採取し、この試料を還元気化原子吸光光度法 によって定量している。

【0034】表1の試験結果から、固化材として高炉セ

メントを用いた固化体は、水銀吸着剤単体に比べて溶出 液中の水銀濃度が低く、固化処理によって水銀の溶出が 抑制されていることは明らかである。

【0035】これに対して、固化材としてポルトランド セメントを用いた固化体、および固化材としてスラグセ メントを用いた固化体では、水銀吸着剤単体に比べて、 逆に水銀の溶出が促進されてしまう現象が見られる。

【0036】一方、溶出液の酸化還元電位は、高炉セメ ントおよびスラグセメントを固化材とした固化体におい て低い値を示しており、これらの固化体が還元性を示し 10 ていることが分かる。

【〇〇37】しかし、スラグセメントを固化材とした固 化体は酸化還元電位が低いものの、硬化剤として水酸化 ナトリウム溶液が添加されているため、水銀吸着剤の溶 解が生じて完全には固化せず、よって水銀の溶出抑制効 果は小さなものとなる。

【0038】この結果から、水銀の溶出を効果的に抑制 しつつ使用済み水銀吸着剤を固定化する上で、ポルトラ ンドセメントと高炉スラグを配合して成る固化材の使用 が最適であることが分かる。

【〇〇39】図3に開示した表2は、ポルトランドセメ ントと高炉スラグとの混合比を変えた各種固化材を用い て作成した固化体に対し、溶出試験を実施した結果を表 している。

【〇〇4〇】この試験では、水銀吸着剤に吸着された水 銀の化学形態を模擬し、試料として硫化水銀の粉末を使 用している。

【0041】また、固化材として、メーカーの異なる高 炉スラグ3種と、ポルトランドセメントとを、5/95、1 0/90、20/80、40/60、65/35、80/20 の各種の重量 30 比で混合したものを用いている。

【0042】固化体は、14.3gの水に35.7gの固化材 を加えて混合し、これに 0.058gの硫化水銀粉末を加え て混合したのち、固形化容器に投入して養生することに より作成している。

【〇〇43】溶出試験は、表1に結果を示した試験と同 じく「産業廃棄物に含まれる金属等の検定方法」環境庁 告示13号(昭和48年2月)に従い、作成した固化体 を粉砕して弱塩酸酸性水溶液中で常温6時間振盪した後 度法により定量している。

【0044】表2から明らかなように、各々の固化体に おける溶出液中の水銀濃度は、高炉スラグのメーカーの 違いによらず、ほぼ同様の測定値を示している。このこ とから、高炉スラグを含んだ固化材における水銀の固定 化効果が、特定な種類の高炉スラグに因るものではな く、高炉スラグが一般的に備えている物性に基づいて発 現するものであることが分かる。

【0045】また、ボルトランドセメントに対する高炉 スラグの配合割合が低い固化材を用いた場合、水銀の溶 50

出量が増大する傾向が見られるものの、高炉スラグとポ ルトランドセメントとの配合割合が重量比で 10/90 ~ 80/20 の範囲では、水銀の溶出量が低い値を示して

【0046】この結果、水銀の溶出を効果的に抑制しつ つ使用済み水銀吸着剤を固定化する上で、高炉スラグと ポルトランドセメントとの配合割合が、重量比で 10/ 90~ 80/20 の範囲にある固化材を使用し得ることが 分かる。

【0047】なお、本発明に関わる水銀吸着剤の固化処 理方法は、天然ガスコンデンセレート(NGL)中から水 銀を除去する吸着除去処理プロセスのみならず、天然ガ ス等のガス状炭化水素や、LNG(液化天然ガス)、灯 油、軽油、ガソリン、ナフサ等の液状炭化水素中から水 銀を除去する場合にも、極めて有効に適用し得ることは 言うまでもない。

[0048]

【発明の効果】以上、詳述した如く、本発明に関わる使 用済み水銀吸着剤の固化処理方法では、ボルトランドセ 20 メントと高炉スラグとを混合して成る固化材を用いて、 水銀を吸着した使用済み水銀吸着剤を固化処理してい

【0049】本発明に関わる使用済み水銀吸着剤の固定 化方法によれば、水銀吸着剤に吸着された水銀を安定に 固定化することできるとともに、アルカリ性の低さから 固化材の固化が阻害されることを抑えられるので、水銀 の不用意な溶出や固化体の強度不足を招くことなく、使 用済みの水銀吸着剤を安定した状態で固定化することが 可能となる。

【0050】また、本発明に関わる使用済み水銀吸着剤 の固定化方法によれば、ボルトランドセメントと高炉ス ラグとを混合して成る固化材は、一般に高炉セメントと して安価に市販されており、日本国内に限らず世界各国 でも容易に入手できるので、簡便かつ安価に使用済み水 銀吸着剤を固化処理することが可能となる。

【0051】さらに、本発明に関わる使用済み水銀吸着 剤の固定化方法によれば、主たる作業は使用済み水銀吸 着剤と固化材および水とを混合するという極めて簡単な ものであるため、使用済みの水銀吸着剤が発生する石油 の液から試料を採取し、この試料を還元気化原子吸光光 40 プラント内であっても、使用済み水銀吸着剤を極めて容 易に固化処理することが可能となる。

【図面の簡単な説明】

【図1】本発明に関わる使用済み水銀吸着剤の固化処理 方法を示す作業フロー図。

【図2】各種の固化材を用いて作成した固化体に対し溶 出試験を実施した結果を示す表1。

【図3】ポルトランドセメントと高炉スラグとの混合割 合を変えた各種固化材を用いて作成した固化体に対し溶 出試験を実施した結果を示す表2。

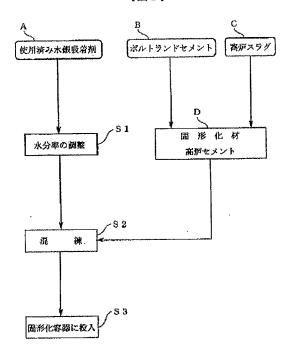
【符号の説明】

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A…使用済み水銀吸着剤、 B…ポルトランドセメント、 C…高炉スラグ、

D…固化材、高炉セメント。

【図1】



【図2】

殺 1	
溶出液中の水果濃度	溶出液の酸化澄元電位
1. 5 ppb	Et = 140 mY
16. 5 ppm	Eh = 50 mV
< 0. 5 ppb	Eli = -93 mV
145 ppb	Eh = -106 mV
	溶出液中の水漿濃度1.5 ppb16.5 ppm< 0.5 ppb

【図3】

₹ 2

固化体(固化材)	高炉スラグ	俗出液中の
(高炉スラダ/ボハトラント゚セナント 比)	メーカー	水鈕邊度(ppm)
5/95 (rt%)	A社	15
10/90 (wt%)	A社	1
20/80 (wU%)	A社	0. 5
	B社	< 0. 1
	C社	< 0. 1
40/60 (wt%)	Αŧt	0.3
	B社	< 0. 1
	C社	< 0. 1
65/35 (wt%)	A社	< 0. 1
	B社	< 0. 1
	C社	< 0.1
80/20 (wt%)	A社	< 0. 1

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